
STRENGTH AND ACID RESISTANCE OF LOW CALCIUM FLY ASH-BASED GEOPOLYMER MORTAR

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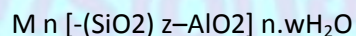
Abstract

The present paper presents fresh and hardened low-calcium fly ash-based geopolymer mortar were studied at different molarity and different curing temperature. Crystallography of fly ash sample with X-ray diffraction (XRD) was analyzed. Tests were carried out on geopolymer mortar specimens of size 70.6 x 70.6 x 70.6 mm. From the test results, it has been observed that as the concentration of sodium hydroxide increases, the compressive strength of geopolymer mortar also increases further, at 800 C the compressive strength of geopolymer mortar specimens were found to be optimal. An experimental program consisting of immersion of geopolymer specimens in 10% solutions of sulfuric acid and nitric acid up to a period of 4 weeks was carried out to evaluate its resistance in terms of visual appearance, change in weight and compressive strength at two and four weeks. Geopolymer mortar samples after exposure did not show any noticeable change in color. The weight loss was eminent in case of exposure to sulfuric acid and minimal in case of exposure to nitric acid. Similar observations were noticed in case of change in compressive strength concluding that geopolymer mortars are more resistant to nitric acid as compared to sulfuric acid.

Keywords: Geopolymer; XRD; compressive strength; weight loss; visual appearance

1. INTRODUCTION

Geopolymers are individuals from the group of inorganic polymers. The microstructure of geopolymer material is shapeless and the concoction creation is like that of normal zeolitic materials. The expression "geopolymer" was instituted by Joseph Davidovits [1-4], in 1978 to characterize the newfound geosynthesis that produces inorganic polymeric materials. Geopolymer materials are created by a sol – gel prepares using alumina and silica oxides enacted by salt hydroxides as well as soluble base silicates. The precipitation procedure is being encouraged by warmth. A three dimensional polymeric chain and ring structure comprising of Si-O-Al-O bonds is framed due the polymerization procedure. The mineral polymers have exact recipe as takes after:



Where: M = alkaline cation for example, potassium, sodium or calcium; the image – demonstrates the nearness of a security, n is the level of polymerization; z is 1, 2 or 3 [1-2]. The schematic development of geopolymer material is shown in Figure 1.

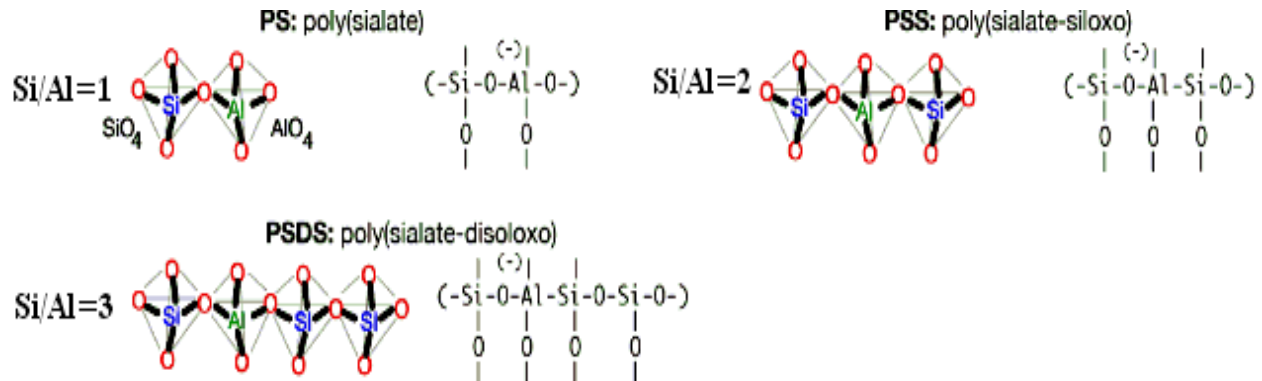


Figure 1: Basic forms of geopolymer (Davidovits 1999)

The properties and uses of geopolymers have been investigated in numerous logical and mechanical controls. Creators in their review presumed that geopolymer have praiseworthy properties, for example, a high early quality, low shrinkage, high imperviousness to solidifying and defrosting, sulfate assault and consumption [3-5]. Geopolymer separated from having superb mechanical properties are likewise impervious to flame and corrosive [6-8] and furthermore does not produce any perilous soluble base total response [9].

Malhotra in his exploration distribution [10] specified that Portland bond discharges around 1.35 billion tons of carbon dioxide in air every year and further remarked that endeavors ought to be made to advance the halfway supplanting of Portland concrete with pozzolanic materials in solid generation. To deliver geopolymer just direct vitality is required. In addition, they are natural amicable materials which amid polymerization handle don't discharge nursery gasses. A few creators expressed that geopolymer or salt actuated bond is an inorganic, alumino-silicate-based material [9, 11, 8]. Fernandez-Jimenez et al. furthermore, Chindaprasirt et al. [12-13] further expressed that the qualities of both Portland bond mortar and geopolymer mortar are comparable to each other.

Numerous scientists [14-19] opined that fly slag is accessible in wealth and henceforth can be considered as an imperative source material for geopolymer. It has been accounted for that geopolymer showed high mechanical properties when arranged with fine fly fiery debris as well as responsive fly powder [20-21]. Encourage, Temuujin et al. [22] revealed that the mechanical properties of fly fiery remains geopolymers are improved by the nearness of mixes like calcium.

Swanepoel et al. also, Alvarez-Ayuso et al. in their work reasoned that, 48 h curing time, might be considered as ideal for geopolymer mortar [23-24]. It was additionally reasoned that at ideal temperature of 800 C and after 48 h of curing period the impact on compressive quality is more noticeable [24]. Then again, a few creators said that expansion in the curing time helps in helping pick up in compressive quality [18-19]. Palmo et al. concentrated the impact of curing temperature, curing time and molarity on geopolymer and presumed that as the curing temperature builds the fly slag initiation quickens [25]. The most critical figure for quality instance of geopolymer at hoisted temperature is fly fiery remains to activator proportion [26].

Davidovits 1999 [9] revealed that for basic respectability geopolymers depend most on alumina-silicate securities and minimum on calcium silicate hydrate securities. Low mass misfortune was additionally detailed by the creator if there should be an occurrence of Metakaoline based geopolymer tests when inundated in 5% arrangement of H₂SO₄ for 4 weeks. Geopolymers arranged from fly cinder are more safe than customary concrete partners when submerged in 5% sulfuric corrosive up to 5 months [8]. A quickened test to survey the toughness of geopolymer cement was directed by Song et al. [27] on geopolymer concrete and inferred that geopolymer cement was sturdy in a 10% arrangement of H₂SO₄ for 56 days. The same has additionally been accounted for by S.E. Wallah and B.V. Rangan [28].

This examination article displays the utilization of fly fiery debris as a source material in the improvement of geopolymer cement rather than concrete. The impact of molarity of NaOH at various curing temperature on the compressive quality parameter of geopolymer mortar has been evaluated. Facilitate, the conduct of geopolymer mortar under acidic condition was measured as far as change in weight, change in compressive quality and visual appearance.

2. EXPERIMENTAL ANALYSIS

2.1. Material properties

In the present investigation the materials used were locally availed and the physical properties investigated through various laboratory tests. Fine low calcium fly ash utilized as a part of the present examination was sourced from Kahalgaon Thermal Power Plant, NTPC India. It has concoction sythesis as substantiated in Table 1. The substance property of fly cinder has been acknowledged in light of the information made accessible from NTPC Kahalgaon Bihar the prerequisites affirm to IS: 3812-1981[29].

Sl. No.	Test Conducted	Observed Values (%)
1	Loss of Ignition	2.32
2	Silica as SiO ₂	42.04
3	Iron as Fe ₂ O ₃	4.40
4	Alumina as Al ₂ O ₃	33.60
5	Calcium as CaO	12.73
6	Magnesium as MgO	0.00
7	Sulphate as SO ₃	0.40
8	Chloride	-
9	Lime Reactivity	4 N/mm ²

Table 1: Chemical properties of fly ash

2.2. Characterization of fly ash

Mineralogical structures of fly ash with X-ray diffraction (XRD) were investigated. Fly ash sample is powdered by grinding in a pestle and was subjected to XRD analysis using a Rigaku Ultima IV system with Cu K α radiation. Scans were run from 20° to 80° with increments of 0.05° and a counting time of 2 s per step. Graph (Figure 2) is obtained from Origin Pro (Plus) software based on the data of XRD of the samples. The graph is compared with JCPDS charts using the X'pert High score (Plus) software by which we get the quantitative analysis of the specimens. From the XRD analysis of fly ash, it is seen that maximum peak is obtained at 26°.

Figure 2 demonstrates XRD example of fly fiery debris as for the most part recognized formless stage and little crystalline stages as quartz, mullite and hematite. Mullite (3Al₂O₃.2SiO₂) is shaped by the aluminum silicate introduce in fly slag further, iron and silica is available as hematite (Fe₂O₃) and quartz mineral separately. Likewise, when 2 θ is between 20 - 35°, it is seen that the undefined stage achieves its most extreme inferring that indistinct stage has silica-like attributes since it is near the greatest pinnacle estimation of quartz precious stone.

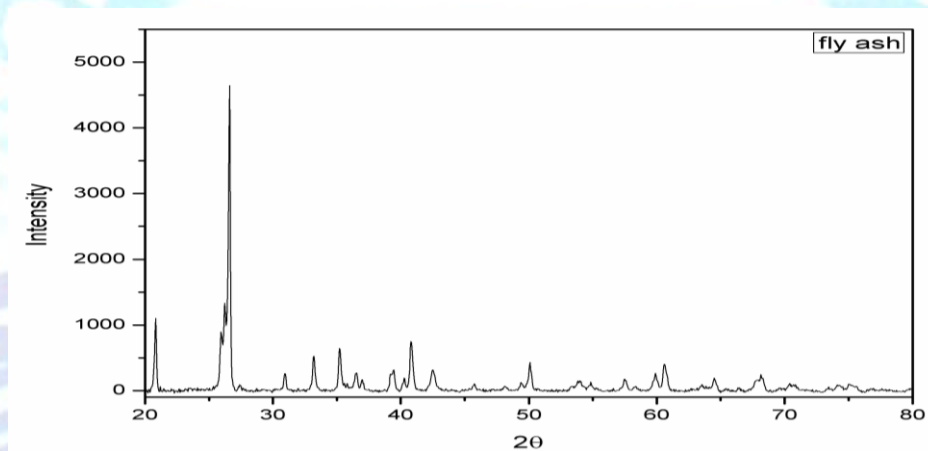


Figure 2: XRD traces of FA. Q= Quartz, M= Mullite, H= Hematite

2.3. Sand

Standard sand confirming to IS: 650-1991[30] confirming to zone II has been used in the present investigation

2.4. Water

To achieve exact morality, distilled water was used all the way through the test procedure.

2.5. Chemical solutions

A combination of sodium silicate arrangement and sodium hydroxide arrangement was utilized as antacid fluid. Both were economically accessible in market. Sodium hydroxide was accessible in pellets frame. Sodium hydroxide pellets were taken and broken down in refined water to accomplish 8, 10, 12,

14, and 16 molarities. The readiness of sodium hydroxide arrangement was prepared 24 h before the utilization in light of the fact that after 36 h it might end to semi strong fluid state as announced in writing. The sodium silicate arrangement was accessible in semisolid frame and was obtained from a nearby provider in mass. The expansion of Na_2SiO_3 permits the amount of the mineral segment to be diminished, and incorporating it in the blend is likewise profitable for speedier solidifying of the fastener.

2.6. Mortar mixes

To achieve the desired molarity sodium hydroxide pellets were taken and dissolved in distilled water. The mass of NaOH solids in a solution varies depending on the concentration of the solution. The fly ash based mortar cubes were of size 70.6mm×70.6mm×70.6mm. As per IS: IS 2250 - 1981 [31] for 1:3 mix proportion for cement sand mortar the quantity of cement comes to 200 g and standard sand comes to 600 g therefore in this study the quantity of fly ash was taken similar to that of cement and fly ash: Standard sand ratio was 1: 3. Further, water was evaluated considering the molarity of sodium hydroxide pellets and extra water was added to maintain consistency of fly ash.

The mass of geopolymer solids is the sum of the mass of fly ash, sodium hydroxide solids and the mass of Na_2SiO_3 (mass of Na_2O and SiO_2 in sodium silicate solution). The mix proportions are presented in Table 2.

S.No	Fly ash(Kg)	Molarity (M)	NaOH (Kg)	Na_2SiO_3 (Kg)	Water (Kg)	Water/Geopolymer solid
1	568	8	20.57	51.43	157.57	0.26
2	568	10	25.72	64.30	168.6	0.28
3	568	12	30.86	77.14	179.56	0.29
4	568	14	36.10	90.00	190.55	0.31
5	568	16	41.14	102.85	201.5	0.32

Table 2: Mix proportioning of Geopolymer mortar

2.7. Test procedure

In all forty five cube sample of fly ash based geopolymer mortar of size 70.6mm×70.6mm×70.6mm were prepared to study the compressive strength parameter. The casting procedure was similar to that of cement mortar. All forty five cubes were immediately placed in universal hot oven along with the mould for curing. Curing temperature was fixed at 600 C, 800 C and 1000 C for different molarity. The moulds after hot oven cuing for 24 h were removed and kept at room temperature for further 24 h. Compressive strength test was conducted in accordance with IS: 4031(Part VI) [32] after 48 h of casting.

To study the effect of exposure to acidic environment, 12 cubes having NaOH concentration of 10 M, 12M and 14 M were casted. The cubes were heat cured at 1000 C for 24 h and further 24 h at room temperature. To study the effect of exposure the cubes after 24 h curing at room temperature were immersed in 10% solution of sulphuric acid and 10% solution of nitric acid for a period of four weeks (Figure 3). The acid resistance of the specimens was evaluated in terms of visual inspection, weight change measurement and compressive strength test after the exposure period of two weeks and four weeks.



Figure 3: Sample immersed in acid

3. RESULTS AND DISCUSSION

3.1. Compressive strength test results

The compressive strength test was applied after 48 h. The NaOH concentration of 8M, 10M, 12M, 14M and 16M were used as variables. The results are presented in Table 3 and shown graphically in Figure 4. The results show that curing temperature is central to the geo-polymerization process of fly ash-based geopolymer mortar. An increase in curing temperature from 60 to 100 °C resulted in an increase in compressive strength of fly ash based geopolymer mortar. Further, no significant enhancement in strength was observed beyond 80°C. However, there was slight decrease in compressive strength at 100°C as compared to 80°C which means that curing at 80°C temperatures gives optimum compressive strength.

Figure 4 and Figure 5 also indicate the development of strength with respect to molarity of activator solution. As molarity increases, the strength increases this may be attributed to the fact that alkaline activator with higher molarity increases the formation of aluminosilicates and geo-polymeric gel, thereby increasing the strength. Chindaprasirt et al. [33] stated that due to filtering of silica and alumina in a highly concentrated NaOH solution, the compressive strength of the samples is expected to increase with an increase in NaOH concentration.

However, there was a minimal decrease in compressive strength at 16 molarity of activator solution as compared to 14 molarity of activator solution. Zuhua et al. in 2009 [34] reported that this might be due

to excess of -OH concentration which further decreases the strength of geopolymer mortar. The polymerization process is disrupted by excess sodium content and can help in formation of sodium carbonate by atmospheric carbonation. This can further lead to viscous mix formation which can further create compaction problem. The same results have been reported by Barbosa et al. [35 - 36].

Another reason may be the effect of water to geopolymer solid ratio or water to fly ash ratio. For the synthesis of geopolymeric material in the initial stage, water content plays a central role. It acts as the medium for dissolution and polymerization of Al and Si precursors. It can be seen from Table 2 that as molarity increases water to Geopolymer solid as well as water to fly ash ratio increases. The results of compressive strengths of geopolymer mortars are also represented in Figure 4 based on water to geopolymer solid ratios varying from 0.26 to 0.32 or water to fly ash ratio varying from 0.28 to 0.35 at 80°C. The results obtained here are in line with the results obtained by the previous researchers [36-37].

S.No	Molarity	Temperature	Water/geopolymer solid	Compressive strength
1	8M	60	0.26	33
2		80	0.26	45
3		100	0.26	50
4	10M	60	0.28	38
5		80	0.28	48
6		100	0.28	52
7	12M	60	0.29	45
8		80	0.29	61
9		100	0.29	64
10	14M	60	0.31	49
11		80	0.31	63
12		100	0.31	67
13	16M	60	0.32	48
14		80	0.32	62
15		100	0.32	65

Table 3: Effect of curing temperature and molarity on compressive strength

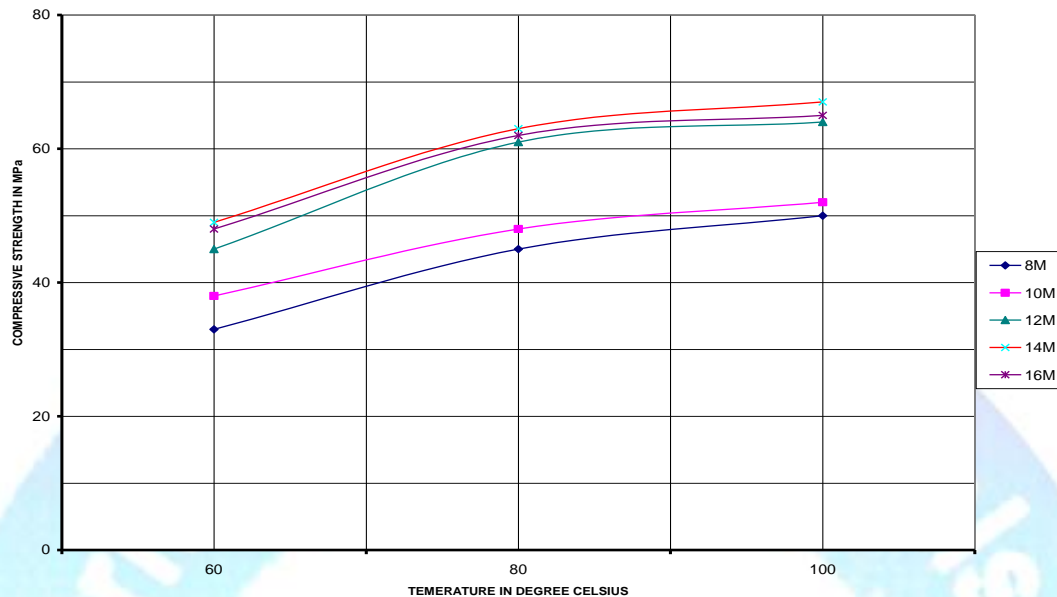


Figure 4: Effect of curing temperature and molarity on compressive strength

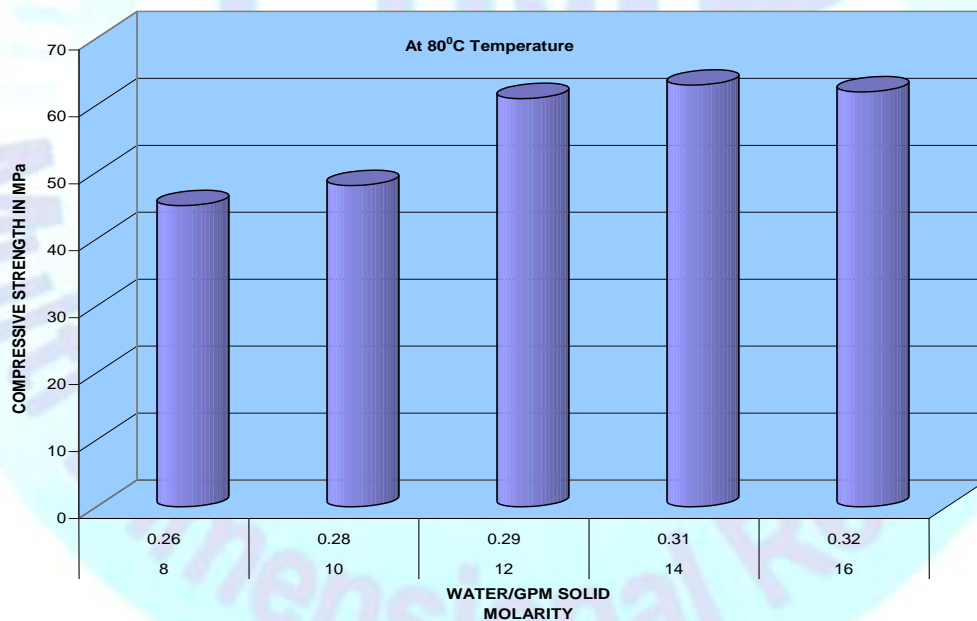


Figure 5: Effect of water to GPM solid and molarity on compressive strength

3.2. Acid resistance test

To evaluate the acid resistance of geopolymer mortar, extra twelve cubes having NaOH concentration of 10M, 12M and 14M were casted. Six cubes each were immersed in 10% solution of sulfuric acid and 10% solution of nitric acid for a period of four weeks. The parameter like change in weight and change in

compressive strength of the mortar specimens were evaluated before and after the exposure period. The specimens showed no noticeable change in color after exposure to acidic solution. The corrosion of the surface increased with time though through visual inspection extent of corrosion among the samples could not be easily differentiated. Photographs of corroded specimen surface taken after four weeks of exposure are presented in Figure 6 and Figure 7 for specimens in sulfuric and nitric acid solution respectively.



Figure 6: Photographs of corroded specimen surface taken after four weeks of exposure in 10% solution of H_2SO_4



Figure 7: Photographs of corroded specimen surface taken after four weeks of exposure in 10% solution of nitric Acid

The geopolymer mortar specimens after exposure to acidic solution remained structurally intact as no cracks were observed and further no change in shape was observed however, leaching was observed on the exposed surface of the specimens which was more evident in case of sulfuric acid solution.

3.2.1. Change in weight

Results of the weight changes for the geopolymer mortars are presented in Table 4. For the specimens immersed in 10% solution of sulfuric acid, weight loss at the end of one week was initially noticed. The percentage decrease in weight was 0.4% for 10M fly ash based geopolymer mortar the percentage decrease was more (0.9%) in case of 12M fly ash base geopolymer mortar and almost 12% in case of 14M fly ash base geopolymer mortar.

Sl.no.	Molarity	Acid	Initial weight in gm	weight after one week in acid	% decrease in weight	weight after two week in acid	% decrease in weight	weight after four week in acid	% decrease in weight
1	10	H ₂ SO ₄	764	761	-0.4	758	-0.8	747	-2.2
2	12		756	749	-0.9	746	-1.3	738	-2.4
3	14		767	758	-1.2	756	-1.4	747	-2.6
1	10	HNO ₃	768	763	-0.7	760	-1.0	754	-1.8
2	12		759	756	-0.4	754	-0.7	749	-1.3
3	14		755	752	-0.4	748	-0.93	744	-1.5

Table 4: Change in weight after acid exposure

Further, the decrease in weight was more prominent after two weeks and after four weeks of exposure. For the specimens immersed in 10% solution of nitric acid fluctuating results of weight changes during the test duration were observed. Contrary to those in sulfuric acid, specimens immersed in 10% nitric acid exhibited lesser weight loss as observed from the results presented in Table 4. Further, as molarity increases the rate of decrease in weight increases in case of geopolymer mortar immersed in 10% solution of sulfuric acid as can be inferred from Figure 8. However, in case of geopolymer mortar immersed in 10% solution of HNO₃ the rate of decrease in weight was almost constant for all three molar solutions as compared to samples immersed in 10% solution of H₂SO₄.

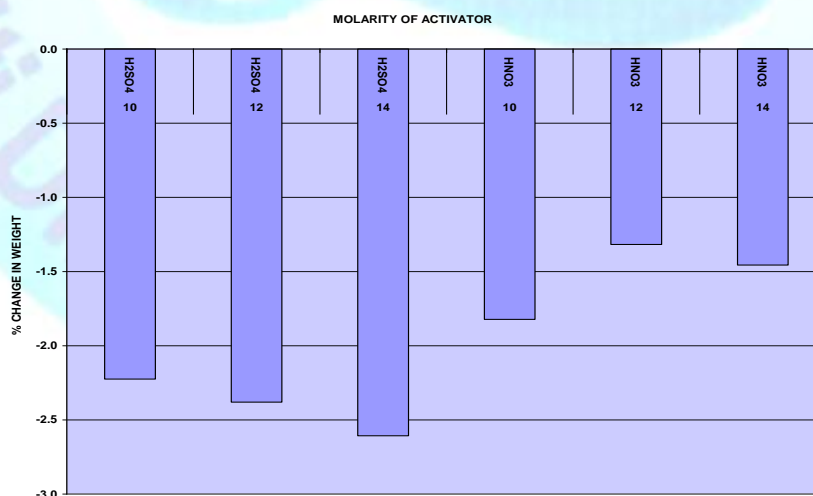


Figure 8: Percentage Change in weight after acid exposure

3.2.2. Change in compressive strength after acid exposure

Table 5 substantiates the compressive strength of geopolymer mortars before and after in sulfuric acid and nitric acid environment. The change in initial and final compressive strength can be interpreted from Figure 9 and Figure 10. From the Figures, it can be inferred that geopolymer mortar specimens of higher molarity showed little loss in strength initially but after four weeks the strength loss was about 8-9% in case of samples immersed in 10% solution of sulfuric acid and around 6% in case of samples immersed in 10% solution of nitric acid.

Sl.no.	Molarity	Acid	Initial strength in MPa	Strength after four weeks in acid	% decrease in strength
1	10	H ₂ SO ₄	52	48	-7.7
2	12		64	58	-9.4
3	14		67	61	-9.0
1	10	HNO ₃	52	49	-5.8
2	12		64	60	-6.3
3	14		67	63	-6.0

Table 5: Change in compressive strength after four weeks in acid

As reported by T. Bakharev [8], the depolymerisation of alumino silicate polymers and liberation of silicic acid, leads to loss in strength of Geopolymer materials in acidic environment. It is also associated to condensation of siliceous polymers and zeolites, which in some cases lead to a significant loss of strength. In acidic environment, geopolymers also deteriorate through crystallization of zeolites and creation of fragile grainy structures.

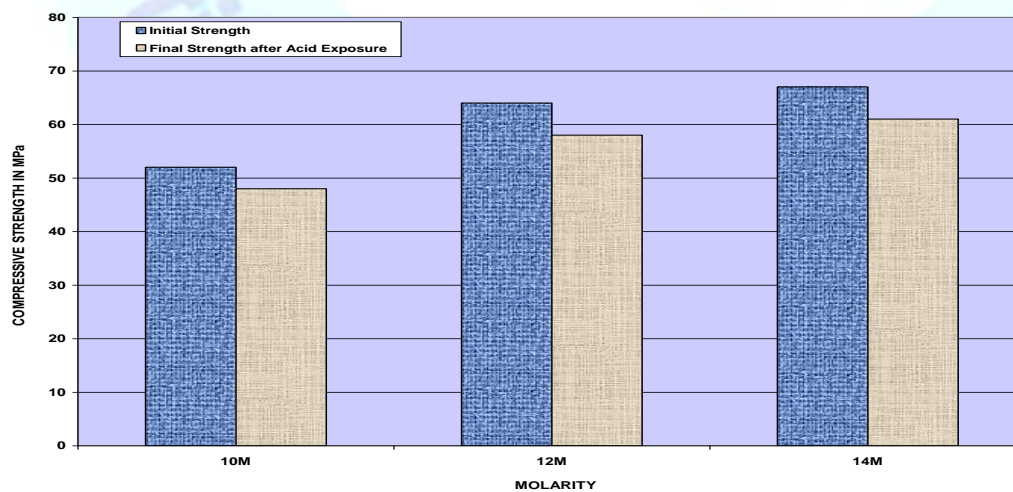


Figure 9: Initial and final compressive strength after H₂SO₄ exposure

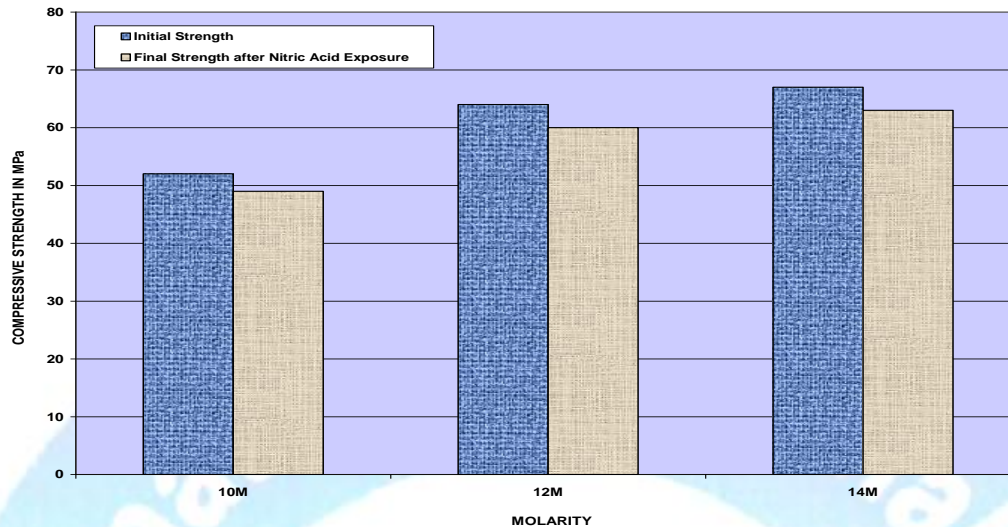


Figure 10: Initial and Final Compressive Strength after Nitric Acid Exposure

4. CONCLUSION

From the test results, it can be concluded that

1. The rate of geopolymerization process increases with the increase in curing temperature. This further leads to accelerated hardening of geopolymer mortar. However, there was no significant improvement observed in the compressive strength of geopolymer mortar when the temperature exceeds 800 C.
2. Increase in molarity of sodium hydroxide solution increase the compressive strength of geopolymer mortar. However at 16M solution of NaOH a slight decrease in compressive strength was observed concluding that 14M is the optimum molarity of NaOH for geopolymer composite.
3. The weight loss was eminent in case of exposure to sulfuric acid and minimal in case of exposure to nitric acid. Similar observations were noticed in case of change in compressive strength concluding that geopolymer mortars are more resistant to nitric acid as compared to sulfuric acid. The findings of the present study shall be useful in determining the applicability of geopolymer materials for use in acid environments

Conflict of Interest

The authors declare that there is no conflicting of interest regarding the publication of this paper.

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